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METALLURGICAL STUDIES WITH POSITRONS

FINAL REPORT

STEPHAN BERKO

MAY 1980

U. S. ARMY RESEARCH OFFICE

DAAG 29 76 G 0320
DAAG 29 77 G 0186

BRANDEIS UNIVERSITY
DEPARTMENT OF PHYSICS
WALTHAM, MA 02254

"Metallurgical Studies with Positrons"

Final Report

15 August 1976 - 31 December 1979

THIS

<u>Introduction</u>. The research performed under Grants DAAG 29 76 G 0320 and DAAG 29 77 G 0186, covering the three year period from 15 August 1976 to 31 December 1979, involved the development and application of the positron annihilation technique to defect studies in metals. The experiments performed during the last three years divide into three categories: (a) the study of the applicability of the simple trapping model to positron annihilation in pure lead as a function of temperature; (b) the study of vacancy-impurity interactions in Pb(Cd) and Pb(Tl) alloys, in order to test the excitonic model of "fast diffusion" of some impurities in Pb; and (c) the design and testing of a multidetector angular correlation setup to perform momentum density measurements in metals with hydrogen, in order to study the role of hydrogen in the electronic structure of pure metals.

1) <u>Precision lifetime measurements in Pb.</u>

The behavior of slow positrons in condensed matter has been the subject of intense experimental and theoretical investigations during the last two decades, partially by the positron group of the Physics Department at Brandeis University. By studying the properties of annihilation quanta one obtains information about the electrons sampled by the positrons injected from a radioactive source into the material under investigation. The defect studies are based on the discovery that thermalized positrons, usually in a delocalized state in a pure metal, can be captured by low density regions of metals, such as vacancies, microvoids, dislocations, as well as macroscopic surfaces. Prior to 1976 very few, if any, high precision experiments were performed to study in detail the sensitivity of the e^+ lifetime technique to various relevant parameters of the theory, in particular temperature. Most of the earlier experiments were performed and analyzed by assuming a priori the applicability of the simple theoretical "trapping model". Our first experiments were involved in studying extensively the question of the precision achievable in vacancy activation energy (enthalpy) measurements. A high sensitivity ultra-stable lifetime apparatus was built that achieved better than 200×10^{-12} sec (FWHM) resolving times with a stability of better than a few picoseconds per day. Lifetimes were obtained in pure Pb and analyzed in a constraint-free way. It was found that vacancy activation energies can be measured to an accuracy of 2 - 5%, provided that the temperature dependence of the positron lifetime in bulk as well as in the vacancy is properly taken into account. This work, performed in collaboration with Professor Turnbull at Harvard University, was described in a paper[1] "A high resolution lifetime study of positron trapping by vacancies in lead" by S. C. Sharma, S. Berko, and W. K. Warburton. In order to be able to analyze the lifetime data to sufficient accuracy a new lifetime analysis technique was developed as described in a paper to Computer Physics Communications[2] by W. K. Warburton. S. C. Sharma, a graduate student working for his Ph.D. on the project, described the details of this work as part of his Ph.D. thesis in 1977. This work indicated that in order to obtain a truly accurate measure of positron trapping in vacancies in lead, the temperature dependence had to be studied below room temperature. A variable temperature cryostat was built for this purpose and the lifetime measurements were extended down to liquid He temperatures. A high resolution Ge spectrometer was also used to study Doppler profiles and the momentum densities in Pb as a function of temperatures. The results of this work were published recently[3] by C.-K. Hu, S. Berko, G. R. Gruzalski, and W. K. Warburton. In order to explain some of the earlier work of S. C. Sharma, a theoretical paper describing the consequences of possible positron pre-thermalization trapping was published by Warburton and Shulman.[4] Our more recent low temperature work renders, however, the pre-thermalization assumption unnecessary.

2) <u>Experiments on Pd(Tl) and Pd(Cd) alloys</u>.

The second phase of our research consisted in the lifetime and Doppler shift studies in Pd(Tl) and Pd(Cd) alloys. At low temperatures ($T < 100\,K°$) a new effect was discovered: Compared to the case in pure Pb, where below the vacancy regime annihilation proceeds from the bulk with a typical linear temperature dependence, in the Pb-Cd system we find evidence for a positron trap - the annihilation parameters are nearly constant with temperature. We interpret this difference between pure Pb and Pb-Cd as being due to micropores induced by the Cd impurity when the Pb-Cd is cooled from the melt. We find that the magnitude of the effect scales between the 4% and 2% samples. In order to test the micropore hypothesis we performed a rolled sample experiment and find that the effect diminishes upon cold-rolling. In the Pb-Tl system the effect does not appear - the solidus-liquidus separation is much smaller than in the Pb-Cd case. Constitutional supercooling would be difficult to avoid in Pb(Cd) because the solute concentration in the solid phase is much less than that in the liquid phase; hence the formation of micropores is likely. Dislocation-pinning by Cd in the Pb(Cd) alloys was also observed. A paper describing the observation of micropores by positron trapping in Pb(Cd) was published[5] by C.-K. Hu, S. Berko, G. R. Gruzalski, and D. Turnbull. The behavior of the positron parameters at elevated temperatures ($T > 100$ K°) indicates that in Pb(Cd) vacancy trapping of positrons is different than in pure Pb. The Pb(Tl) samples gave the same behavior as pure Pb. The first results of these measurements were described in a short paper.[3] Cd is known to be a "fast diffuser" in Pb, while Tl is not; it is therefore perhaps feasible to assign the observed difference between Pb(Cd) and pure Pb trapping to atomic exciton formation by Cd. On the other hand other effects such as a dependence on solute concentration of the delocalized positron annihilation rate may also be responsible for this behavior. Further experiments on single crystals with high precision angular correlation studies will be necessary to further clarify the interpretation of these results.

3) <u>Angular correlation of annihilation radiation studies</u>.

In order to study the importance of hydrogen on the electronic structure of metals, we proposed a study of momentum density measurements in the Ta-H system by the angular correlation of annihilation radiation technique (ACAR). As part of this project as well as for the study of Fermi surfaces in alloys, a research program supported by a grant from the National Science Foundation, a new high precision multidetector two-dimensional ACAR apparatus was designed and built during the last three years. The new machine, consisting of 64 detectors, allows the high precision measurement of the electronic momentum density in metals. A review of this apparatus and the first measurements on pure metals was published by S. Berko.[6] Unfortunately Professor S. C. Moss of the University of Houston, with whom we are collaborating on the topic of hydrogen in Ta, was able to deliver the hydrogen loaded Ta single crystals only in the fall of 1979; we have obtained preliminary ACAR measurements in pure Ta and in a Ta-18 at.% H sample. The preliminary results indicate that the presence of electrons from H renders the Fermi surface of Ta quite diffuse; more experiments on intermediate H concentrations are in progress. A brief general review of the positron annihilation technique in metal studies was recently published[7] by S. Berko in "Scripta Metallurgica".

Publications List

1. "A high resolution lifetime study of positron trapping by vacancies in lead," S. C. Sharma, S. Berko, and W. K. Warburton, Phys. Lett. 58A, 405 (1976).

2. "DBLCON: A version of positronfit with non-gaussian prompt for analysing positron lifetime spectra," W. K. Warburton, Comp. Phys. Comm. 13, 371 (1978).

3. "Positron annihilation lifetime and Doppler profile studies in Pb, Pb(Tl), and Pb(Cd)," C.-K. Hu, S. Berko, G. R. Gruzalski, and W. K. Warburton, Proc. 5th Int. Conf. Positron Annihilation (Japan, 1979), p. 231.

4. "A model allowing pre-thermalization trapping of positrons," W. K. Warburton and M. A. Shulman, Phys. Lett. 60A, 448 (1977).

5. "Evidence for microporosity and dislocations in Pb(Cd) Alloys from positron annihilation studies," C.-K. Hu, S. Berko, G. R. Gruzalski, and D. Turnbull, Solid St. Comm. 31, 65 (1979).

6. "Two-dimensional angular correlation of annihilation radiation experiments," S. Berko, Proc. 5th Int. Conf. Positron Annihilation (Japan, 1979), p. 65.

7. "Positron annihilation experiments in metals; electronic structure and Fermi surface studies," S. Berko, Scripta Metallurgica 14, 23 (1980).

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Dr. G. R. Gruzalski, Research Associate, 1978-79 (funded by Harvard University)

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Dr. G. M. Beardsley, Research Associate, 6/1/77 - 7/31/77 (supported by ARO grant)

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C.-K. Hu, 9/1/76 - 8/31/78

W. S. Farmer, 1/15/78 - 8/14/78

Ph.D. Theses

1) S. C. Sharma, 1977, "High Resolution Lifetime Study of Positron Trapping by Vacancies in Pb and Pb-4 at.% Cd" - presently Assistant Professor, University of Texas at Arlington, Texas

2) C.-K. Hu, 1979, "Studies of Defects in Pb, Pb(Tl), and Pb(Cd) by Positron Annihilation Lifetime and Doppler Profile Measurements" - presently Research Associate, Rensselaer Polytechnic Institute, with Professor H. Huntington